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Inventor(s) : TANAKA et al.
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For : ALLOYED MOLTEN ZINC PLATED STEEL SHEET AND
PROCESS OF PRODUCTION OF SAME

Commissioner for Patents
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DECLARATION UNDER 37 C.F.R. §1.132

S I R:

I, Koki TANAKA, a citizen of Japan, declare as follows:

I. BACKGROUND

(1). I am a co-inventor of the invention(s) disclosed and claimed in the above-identified patent application.

(2). I am an employee of Nippon Steel Corporation, Tokyo, Japan. Nippon Steel Corporation, Tokyo, Japan, in a joint assignee of the above-identified patent application. The other joint assignee of the above-identified patent application is USINOR, Pateaux, France.

(3). I graduated from Nagoya University, Japan in March 1982 and I graduated from Nagoya University, Japan in the Master Course Engineering Faculty in

March 1984. I received a Doctor Degree of Engineering from Osaka University, Japan in 1998.

(4). Since 1984, I have been employed by Nippon Steel Corporation, Tokyo, Japan at the Technical Development Bureau engaged in research with respect to materials characterization.

(5). I can read and understand the English language. I can read and understand the Japanese language.

(6). I have read and understand the specification, claims and drawing of the above-identified patent application, and specifically claims 1 to 3 under consideration in the above-identified patent application. I have read and understand the prior art of record in the above-identified patent application and specifically Japan 2000 - 290730 and Japan No. 2003 - 055751.

(7). In the Office Action mailed June 4, 2008, claims 1 to 3 were rejected under 35 U.S.C. §102(b) as being anticipated by Japan No. 2000-290730 to Suzuki et al.

(8). In the Office Action mailed June 4, 2008, claim 3 was rejected under 35 U.S.C. §103(a) as being unpatentable over Japan No. 2000-290730 to Suzuki et al. in view of Japan No. 2003-055751 to Fujita et al. (incorrectly identified in the Office Action mailed June 4, 2008 as Japan No. 2000-005751).

II. Japan No. 2000-290730 ("JP '730")

(1). The product obtained by the present invention is different from the product obtained by JP '730 because of the difference in the production process.

(2). An important difference from the present invention is that JP '730 does not have the feature of "an average diameter of the particle size of the oxide is 0.01 - 1 μm " for the oxide particles in the plating layer which is an important feature of the present invention as defined in claim 1.

(3). The production process disclosed in JP '730 is: hot rolling → removing mill scale (black scale) → cold rolling → CAL (annealing in the specified atmosphere defined in Formula 1) → pickling → CGL (annealing in a reducing furnace in the specified atmosphere defined in Formula 2) → galvanizing in a zinc bath.

(4). The production process defined in the present invention is: hot rolling → cold rolling → CGL (annealing in the specified atmosphere defined in claim 4 which is currently withdrawn from consideration) → galvanizing in a zinc bath → galvannealing.

(5). In JP '730, the fine inner oxide layer having several microns thickness after CAL is formed only within the range of Formula 1 of JP '730. Otherwise, the inner oxide layer does not fine in JP '730 in the case of being outside the range of Formula 1 of JP '730. (JP '730 [0007]).

This inner oxide oxide layer of JP '730 improves platability in CGL because the inner oxide layer is formed during the heating step because of the high oxygen potential, and this inner oxide layer remains just before the plating step, and prevents a dispersion of dissolved Si and Mn existing in the steel sheet to the steel sheet surface. As a result, surface concentration is restrained and platability is improved. (JP '730 [0018]).

(6). Surface concentrates containing Si and Mn are removed in JP '730 by pickling. (JP '730 [0042]).

(7). CGL annealing atmosphere: It is desirable in JP '730 that the CGL annealing atmosphere is a sufficient reduction atmosphere for securing platability by means of reduction of the oxide film formed on the steel sheet surface after pickling. (JP '730 [0047]).

(8). That is: the inner oxide layer of JP '730 formed in II. (5). is grown by absorption of Si, Mn dispersed from the inside of the steel sheet when restrained by a

dispersion of dissolved Si and Mn existing in the inside of the steel sheet going to the steel sheet surface layer during CGL annealing, which is called "Ostwald growth".

(9). The oxide particles having a particle diameter of more than $0.01\ \mu\text{m}$ and less than $1\ \mu\text{m}$, as defined in the present invention, are very energetically unstable under the condition of a fine inner oxide layer having several microns thickness being formed, even if the inner oxide layer is formed, and then such oxide particles disappear during Ostwald growth of the inner oxide layer.

(10). As mentioned above, JP '730 aims to remove dissolved Si and dissolved Mn remaining in the surface layer by pickling. In addition, the CGL atmosphere in JP 730 is fixed for a sufficient reduction atmosphere defined in Formula (2) of JP '730. Therefore, oxide particles are not newly formed in the inner oxide layer formed during CAL. The inner oxide layer formed during CAL is grown by absorption of Si, Mn dispersed from the inside of the steel sheet when restraining a dispersion of dissolved Si and Mn existing in the inside of the steel sheet going to the steel sheet surface layer during CGL annealing.

(11). The oxide particles having a particle diameter of more than $0.01\ \mu\text{m}$ and less than $1\ \mu\text{m}$, as defined in the present invention, are very energetically unstable under the condition of a fine inner oxide layer having several microns thickness being formed, even if the inner oxide layer is formed, and then such oxide particles disappear during Ostwald growth of the inner oxide layer.

(12). According to the present invention, oxide particles having a diameter of more than $0.01\ \mu\text{m}$ and less than $1\ \mu\text{m}$ are formed on the steel sheet surface in CGL, and these oxide particles move to the plated layer. Then Fe-Zn alloying is promoted by means of a reaction of these oxide particles contained in the plated layer. In JP '730, there is no formation of oxide particles having a diameter of more than $0.01\ \mu\text{m}$ and less than $1\ \mu\text{m}$ on the steel sheet surface. There is no movement of the inner oxide layer.

(13). It is clear that the inner oxide layer formed by CAL in JP '730 does not move to plated layer by the following experiment conducted under my supervision.

(14). An experiment under my supervision was conducted under the same conditions of JP '730 to obtain a plated steel sheet. The structure of the plated steel sheet obtained by the experiment was observed at the cross-sectional area of the thus obtained plated steel sheet. The following conditions were present in the experiment.

(i). The steel sheet contains Si: 1.2 mass %, Mn: 1.6 mass %.

(ii). Annealing atmosphere: $\text{H}_2\text{O}/\text{H}_2$: 0.1, H_2 : 5%, N_2 : 95%, annealing at 800°C for 60 seconds.

(This experimental condition was the same condition shown in Fig. 2 of JP '730).

(iii). Pickling was not done because this experiment was carried out in a laboratory.

(iv). Then annealing was carried under the following conditions.

Atmosphere: $\text{H}_2\text{O}/\text{H}_2$: 2×10^{-3} , H_2 : 5%, N_2 : 95%, annealing at 800°C for 60 seconds → cooling to 470°C → galvanizing → heating at 450°C for 60 seconds → alloying treatment.

(This experimental condition was the same condition shown in the Fig. 3 of JP '730).

(v). The cross-sectional area including the plating layer was polished and Si distribution was observed by EPMA with the following observations made.

(a). As shown in Fig. 1 attached hereto, although the inner oxide layer was formed on the surface of the steel sheet, oxide particles were not found in the plating layer.

(b). Further, the inner oxide layer was formed as a film state having several microns thickness. The experiment conducted under my supervision in accordance with the

teachings of JP '730 confirmed that oxide particles were not taken into the plating layer during alloying treatment because the zinc dispersion is prevented by the inner oxide layer.

(15). The experiment conducted under my supervision confirmed that the subject matter of independent claim 1, and claims 2 and 3 dependent thereon, is not disclosed or suggested by the teachings of Japan No. 2000-290730.

III. Japan No. 2000 - 055751 ("JP '751")

(1). The technology disclosed in the JP '751 relates to a high strength, hot dip galvanized steel sheet having excellent plating adhesion at high working and excellent ductility.

(2). JP '751 discloses that Mn is an easier formed oxide film than Al which is added to the Zn bath, and increases reaction with the Si system oxide film formed on the steel sheet surface. (JP '751 [0017]).

(3). This means that the Si system oxide film in JP '751 is formed on the steel sheet surface before plating. On the other hand, according to the present invention, although oxide particles having a diameter of more than 0.01 μm and less than 1 μm exist underneath up to 1 μm of the steel sheet, there is no Si system oxide film formed on the steel sheet surface.

(4). The steel sheet of JP '751 is annealed at a temperature calculated from the Ac_1 and Ac_3 points in an atmosphere composed of 10% H_2 - N_2 . This means that the steel sheet is retained in 10% H_2 - N_2 atmosphere. (JP '751 [0065]). According to the present invention, steam is introduced into N_2 gas with 10% of H_2 gas, and the amount of steam is controlled and further there is control of the ratio of $\text{PH}_2\text{O}/\text{PH}_2$. The annealing atmosphere of the JP '751 is quite similar to the processing conditions of Comparative Examples Nos. 2, 5, 6 and 8 in Table 2 at page 17 of the present specification.

(5). The Test Material Code and Treatment Condition Nos. NA-5, NA-8, A-5, A-8, B-2, B-5, B-6, C-2, C-5, C-6, D-5, D-6, E-5 and E-6 in the Comparative Examples in Table 3 at page 19 of the specification of the present application correspond to the comparative processing conditions Nos. 2, 5, 6 and 8 in Table 2.

(6). In these comparative examples of Table 3, ND is marked because it is impossible to detect the oxide particles in the plating layer. See end of Table 3 at page 20 of the specification. This means that there are no oxide particles in the plating layer in Comparative Examples of the present application which are similar to the disclosure of JP '751.

(7). JP '751 clearly discloses that the result of JP '751 can be obtained by an appropriate amount of Mn add to the Zn plating bath, and Mn more easily forms oxide film than Al which is added to the Zn plating bath, and Mn increases reaction with the Si system oxide film formed on the steel sheet surface for preventing plating defects. (JP '751 [0017]). This is quite different from the characteristic features of the present invention. JP '751 does not cure the defects of the disclosure of JP '730 with respect to the present invention defined in independent claim 1 and dependent claims 2 and 3.

IV. Declaration

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

Koki Tanaka
Koki TANAKA

March 16, 2009
Date